

PATENT  
CASE 4233C3

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Donald B. Appleby et al. : Group Art Unit: 1211  
Serial No.: 08/360,184 : Examiner: E. White  
Filed: December 20, 1994 :  
For: Polyol Polyester Synthesis

DECLARATION UNDER 37 C.F.R. §1.608 OF MILTON C. McMULLEN

Assistant Commissioner for Patents  
Washington, DC 20231

Dear Sir:

I, MILTON C. McMULLEN, declare that:

1. I am employed by the assignee of the present application, The Procter & Gamble Company, and have been working for The Procter & Gamble Company continuously since 1974.
2. From 1985 to 1989 I worked as a technician on the sucrose polyester synthesis project in which sucrose and fatty acid methyl ester were reacted to form sucrose fatty acid esters.

I was responsible for analyzing samples of reactor streams from pilot plant continuous sucrose polyester production processes according to established procedures.

3. I participated in the pilot plant continuous sucrose polyester production process designated P90117 which was conducted from January 18, 1989, to January 27, 1989. The P90117 pilot plant continuous sucrose polyester production process, including operation of the continuous sucrose polyester synthesis equipment, sample collection and data preparation, was performed under the direction and control of Mr. Scott Pearson.

4. During the P90117 pilot plant continuous sucrose polyester production process, I determined the weight percentage of unreacted sucrose in samples taken from the recirculation loop of the initial reactor, i.e., the first reactor R600, according to the established procedure described below, and I accurately recorded the determined weight percentage of unreacted sucrose in the samples on data sheets which were maintained in the pilot plant for the purpose of recording such data. The data which I accurately recorded included the date and time at which a sample was taken, the reactor recirculation loop from which the sample was taken, and the determined weight percentage of unreacted sucrose in the sample. I also initialed the recorded data for each sample which I analyzed.

5. I have examined Exhibit 9, and I confirm that Exhibit 9 comprises accurate copies of the data sheets from the P90117 pilot plan continuous process on which I accurately recorded the date and time at which a sample was taken, the reactor recirculation loop from which the

sample was taken, and the determined weight percentage of unreacted sucrose in the sample. The entries bearing my initials (MM) indicate those samples which I analyzed as described herein and for which I recorded the data as described herein.

6. The established procedure for determining the weight percentage of unreacted sucrose in a sample during the P90117 pilot plant continuous process employed a YSI-27 Industrial Analyzer. In the analyzer, the sucrose is enzymatically converted to glucose, the glucose is enzymatically oxidized to generate hydrogen peroxide, and the hydrogen peroxide is electrochemically oxidized, yielding a response proportional to the sucrose concentration. According to the established procedure, the YSI-27 Industrial Analyzer was calibrated using standards of 1000 and 2000 mg/dl. A weighed amount of the liquid reactor sample was mixed with about 95 milliliters of warm tap water (about 160°F) and the diluted sample was stirred at a temperature of from about 150°F to about 160°F for about 5 minutes. The sample was removed from the heat source and an oil layer was allowed to rise to the surface. A portion of the aqueous layer was collected and injected into the YSI-27 analyzer. The analyzer output was read and the percentage of unreacted sucrose in the sample was determined using the following equation:

$$\text{Wt. \% Unreacted Sucrose} = \frac{\text{Instrument Read Out (mg/dl)}}{\text{Sample Weight (g)} \times 10} .$$

7. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States

Code and that such willful false statements may jeopardize the validity of the present application or any patent issued thereon.

Respectfully submitted,

By: Milton C. McMullen  
Milton C. McMullen

Date: 3-30-95

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